Laboratory, Richland, WA). In a mixture experiment, the response variable depends on the proportions of the components, which must sum to one. Because of this constraint, standard polynomial models cannot be used to analyze mixture experiment data. To get around this, some researchers ignore one of the components and use standard polynomial models in the remaining components. Because the component proportions must sum to one, the ignored component (referred to as the "slack variable" (SV)) makes up the remaining proportion of the mixture. In the literature, there have been many examples of researchers using the SV approach instead of a mixture approach. We have analyzed several of these examples using both approaches. For screening examples, we fit full linear models and identified which components were important using both approaches. In six screening examples, the mixture approach revealed that the SV had a significant effect on the response. For the quadratic examples, we used stepwise regression to develop reduced quadratic models for the SV approach, and partial quadratic mixture (PQM) models for the mixture approach. In three examples, the PQM models identified the SV and/or one of its quadratic blending terms as having a significant effect on the response variable. Hence, by completely ignoring a component's effect on the response, SV analysis carries an inherent risk of wrong conclusions. There are fewer possible reduced quadratic SV models than possible PQM models because the reduced quadratic models are a subset of the class of PQM models. As a result, the PQM models will always fit the data as well as, or better than, the best reduced quadratic SV model. Our research concludes that it is better to analyze mixture experiments using methods specifically developed for them instead of using standard methods with the SV approach.

Planning for the Future: Updating Energy Forecasting Techniques. Catherine Sampson (*Western Washington University, Bellingham, WA*); Todd Samuel (*Pacific Northwest National Laboratory, Richland, WA*). Every year the Energy Information Administration publishes a document known as the Annual Energy Outlook (AEO), which provides analysis and forecasts of world energy markets through the year 2025. The results of this publication are used in the decision making processes of policy makers and public and private investors alike, and are the most comprehensive energy forecasts currently available. However, the National Energy Modeling System (NEMS), the program used to produce these forecasts, is riddled with minor flaws that may have major impacts on the applications of the AEO results. Though built using a detailed, modular structure, NEMS can only be run deterministically on a scenario by scenario basis. Further, NEMS models display an asymmetric loss function, making the results ambiguous to users whose loss function is not identical to that of modelers'. That is why the Department of Energy and the National Renewable Energy Laboratory are commissioning a new model for forecasting energy use. Known as the Stochastic Energy Deployment System (SEDS), this model will improve on current energy modeling mechanisms by providing for full probabilistic treatment of uncertainties. SEDS is in development under experts from several national laboratories, and will be designed on Analytica, a commercially available software package offering a user-friendly format. Hopefully, SEDS will be able to provide forecasts with better representations of the various possible futures of energy markets than are currently available.

Testing the Multiwavelet Representation of Functions. Owen Worley (*Dartmouth College, Hanover, NH*); George Fann (*Oak Ridge National Laboratory, Oak Ridge, TN*). The multiwavelet transformation of functions is one of the most promising methods for analyzing and performing operations on them. The multiwavelet expansion represents functions in terms of a basis of discontinuous multiwavelet functions, which are nonzero over a unit domain. In particular, representations of the Green's function of operators of partial differential equations, constructed in multiwavelet bases, can be proven, in many cases, to be sparse and nearly diagonal. Thus, the computational complexity of the application of these operators is linear or nearly linear with respect to the problem size, and so is very attractive as a method of solution. As in the development of any complex software, testing must be done to assure that the transformed function behaves correctly with respect to basic algebraic and calculus operations. This testing is done by creating and inputting a variety of functions into a program, calculating the multiwavelet representation of these functions, applying a number of operators to both the original and transformed functions, and comparing the results, demonstrating that operating on the multiwavelet representation is numerically stable and achieves the required accuracy. Said testing demonstrated that the transformed functions do behave correctly with respect to the algebra and calculus operations tested to a high degree of accuracy. Also, as the tests were scaled to higher numbers of processors, the completion times decreased in a smooth log curve. These results were expected, but in obtaining them, debugging was performed and problems were identified and worked around. Further testing should be performed on functions represented with a higher wavelet order, and testing should generally be done as the code is modified and improved. The Joule program, written by George Fann and Robert Harrison, performs the multiwavelet transformation which is tested. The paper, Adaptive Solution of Partial Differential Equations in Multiwavelet Bases, by B. Alpert et al, provides background on multiwavelet transformations.

Materials Sciences

The Effects of Amorphous Carbon Coating of Standard Modified Graphite and Soft Carbon on Anode Properties for Li Ion Batteries. David Abram (*University of Illinois, Urbana, IL*); Jun Liu (*Argonne National Laboratory, Argonne, IL*). Hybrid electric vehicles are gaining popularity to increase fuel efficiency and lessen dependency on oil. The battery used is predominantly nickel metal hydride, but there is a push to use Li ion batteries due primarily to their higher gravimetric and volumetric energy density. Improvements in anode quality can be made, and six Hitachi anodes consisting of blank soft carbon and surface modified graphite as well as the same materials with an amorphous carbon coating were examined. Formation cycles were run for half cells against lithium and full cells against $LiNi_1/3Co_1/3Mn_1/3O_2$. Hybrid pulse power characterization tests were run to determine the area specific impedance at various depths of discharge for the full cells. The half cell formation cycles showed that the charge capacity neared the theoretical capacity limit for graphite of 372 mAh/g while the capacity was much lower for soft carbon at around 220 mAh/g. The coating increased the capacity by 4% and reduced the area specific impedance by 36% for the soft carbon. The effects at the same particle size of 20 microns were inconclusive for the graphite, but there was a 25% decrease in area specific impedance when the particle size was decreased. The effect of the coating on the performance of the soft carbon was visibly beneficial while the effects for the graphite may have been due more to particle size. A new test involving a 10 micron graphite blank and a larger electrode area will be done in the future to closer examine the phenomena.

High Activity Fuel Cell Catalysts via Mesoporous Nanocomposite Polymers. Gregory Baker (*Pennsylvania State University, University Park, PA*); Eric Cochran (*Ames Laboratory, Ames, IA*). Hydrogen fuel cells have the potential to improve the way we propel our vehicles. Catalyst particles within cathode catalyst layer (CCL) promote the reaction of protons, electrons, and oxygen, producing water, which must be removed. The current CCL design suffers from poor mass transport properties, limiting the efficiency of the present-day hydrogen fuel cell. The current structure is a combination of different materials put together to achieve these goals but lacks the order needed to achieve the desired efficiency. In this research, we investigated the development of a novel CCL design that will significantly increase the transport of reactants to active catalyst sites. The first step towards this design was the synthesis of a new catalyst support, based on single-wall carbon nanotubes (SWCNTs), that integrates electon and proton conductivity into a single particle. First, pristine SWCNTs were functionalized with an aniline derivative compound using a solvent free technique. Then, azide-terminated polystyrene was "click-coupled" to the alkyne group. This SWCNT-graft-polystyrene was then sulfonated; this created negatively charged regions in the polymer, which facilitated the deposition of platinum nanoparticles through the reduction of platinum salts. After sulfonation the polymer is also proton-conductive. Thermogravimetric analysis analyzed the effectiveness of the grafting reactions. Nuclear magnetic resonance spectroscopy demonstrated that the proper products were prepared during the synthesis of the aniline derivative and also to ensure that azide terminated polystyrene was achieved. Transmission electron microscopy determined the effectiveness of the decoration with platinum.

Oxidation Characterization of Chromium Tungsten Niobium Superalloys. Amanda Bastidos (*University of Texas at El Paso, El Paso, TX*); Ken Natesan (*Argonne National Laboratory, Argonne, IL*). The past 50 years of metallurgical and materials engineering has brought about much more technologically advanced materials and alloys. One of the dilemmas with current alloys used in high temperature environments is the temperature limit seems to peak at 1,000°C. The overall goal of this research project is to characterize the oxidation layers of a chromium tungsten niobium (CrWNb) superalloy in short term oxidation (STO) experiments and long term oxidation (LTO) experiments in high temperature environments (700–1,400°C).

The STO samples undergo heat treatment in the furnace for 24 hours from 700–1,400°C in increments of 100 degrees and subsequently furnace cooled. Plots of weight gain per total surface area vs. oxidation temperature are obtained from the data. The LTO experiment follows the same set of procedures as the STO with the change from 24 hours to 168 hours; plots of weight gain per total surface area vs. oxidation time are obtained from the data. At temperatures between 700–900°C, the samples were primarily powder as opposed to temperatures 1,000°C and above where the samples maintained their solid forms. To characterize the oxidation layers of the CrWNb samples, scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS) were be performed. If the research is successful, the application of this superalloy will be in gas turbines, combustion liners, fossil-fuel fired combustion systems, turbine blades, etc.

Photovoltaic Properties of Epitaxially Grown Bismuth Ferrite. *Sourav Basu* (***University of California at Berkeley, Berkeley, CA***); *Ramamoorthy Ramesh* (***Lawrence Berkeley National Laboratory, Berkley, CA***). Photovoltaic properties of epitaxially grown (111) $BiFeO_3$ (a ferroelectric material) were investigated under a 100 mW/cm^2 solar simulator light source and tunable halogen source. Photo-excited carriers were extracted through transparent Indium Tin Oxide (ITO) top electrodes. Carrier migration was through-film, in the direction of intrinsic polarization. Current densities observed under solar spectrum at no applied field exceeded 1 mA/cm^2 for certain samples, much higher than previously observed in photovoltaic ferroelectric materials. All films were grown by Pulsed Laser Deposition (PLD). Annealing of samples in varying partial pressures of oxygen was conducted post-deposition to introduce oxygen vacancies and consequently improve the conductivity of the $BiFeO_3$ films. The magnitude of the photocurrent was observed to be directly proportional to the conductivity of the migration pathway. Traditionally, photovoltaic ferroelectric materials have been prevented from being used in energy conversion primarily because of their low current density output. By observing current densities in excess of 1 mA/cm^2 (on the order of magnitude of current densities produced by many photovoltaic semiconductors), we demonstrate the possibility for the use of $BiFeO_3$ in direct energy conversion. In addition, preliminary results involving wavelength dependence of photocurrent indicate the presence of the absorption edge near a wavelength corresponding to $BiFeO_3$'s theoretically predicted bandgap. In addition, future research involving the multiferroic properties of $BiFeO_3$ could present the possibility of optically controlled magnetization.

Doping of TiO_2 (with C and S) for Visible-light Absorption. *Rohit Biyani* (***Washington State University, Pullman, WA***); *Theva Thevuthasan* (***Pacific Northwest National Laboratory, Richland, WA***). Hydrogen proves to be a very clean and efficient fuel source; unfortunately obtaining large quantities of it is not cheap. One of the potential methods of obtaining hydrogen involves photocatalytic splitting of water molecules with Ultraviolet light using Titanium Oxide (TiO_2) as the catalyst. However, an Ultraviolet light source is expensive; a cost efficient substitute is visible light or sunlight. Band engineering of TiO_2 single crystals with appropriate dopants can facilitate photocatalytic activity for visible-light absorption. Anion dopants such as nitrogen, carbon and sulfur have been implanted into the TiO_2 substrate, and it has been demonstrated that some of these implanted materials can absorb visible light for photochemistry. Our overall objective is to develop fundamental scientific understanding about the mechanisms associated with photochemistry in these materials. In this study, we used ion accelerator to implant C and S dopants in single crystal TiO_2 (110) samples and to investigate the structural changes in the materials. The samples were implanted at various temperatures and doses, the lattice site location of the dopants were analyzed using ion beam capabilities. The implanted TiO_2 substrates were characterized using Nuclear Reaction Analysis (NRA), Rutherford Backscattering Spectrometry (RBS), and Proton Induced X-ray Emission (PIXE) methods in random and channeling geometries. Although PIXE is extremely sensitive to trace elements, S quantification could not be made using PIXE due to the domination of the signal from Ti. The signal from S could not be collected without too many uncertainties. The RBS along channeling and random geometries showed that the greater the temperature of implantation, the less the damage was on the surface. Also a lower carbon dose at a high temperature of 900°C shows some substitutional behavior of the carbon atoms replacing possibly oxygen in the TiO_2 lattice. The samples were finally annealed at 900°C in air for several hours and as expected, the implantation damage was significantly reduced in the annealed samples. In addition, the angular yield curves show that the C atoms moved to interstitial positions during annealing. Further experiments are necessary to understand the influence of C dose on this behavior of C location in TiO_2 lattice.

High Storage Density Capacitors Fabricated Using Atomic Layer Deposition. *Andrea Blumentritt* (***LeTourneau University, Longview, TX***); *Jeffrey Elam* (***Argonne National Laboratory, Argonne, IL***). Capacitors are devices which store charge and consist of an insulator between two conducting layers. Capacitors with high energy storage densities are extremely attractive as replacements for conventional batteries in electric and hybrid vehicles. This project sought to design and build high storage density capacitors with small insulator thicknesses and large surface areas-two factors that increase capacitance. The conducting and insulating layers were deposited by atomic layer deposition (ALD), a thin-film process which coats atomic monolayers of material through sequential self-limiting reactions. ALD is extremely useful for this application because it can deposit very uniform layers on high surface area substrates. Anodic aluminum oxide (AAO) membranes were used as the high surface area substrate material. Circular AAO membranes with a diameter of 13 mm and a thickness of 60 μm were used. These contained 200 nm pores which closed down to 20 nm at one end of the disc and had a pore density of 109/cm^2. The AAOs were placed in the ALD reactor and coated with layers of aluminum-doped zinc oxide (AZO, a conductor), aluminum oxide (Al_2O_3, an insulator), and again with AZO. A piece of silicon was also placed in the reactor as a baseline for measuring film thickness. After analysis by an ellipsometer, thicknesses of the final trial were recorded to be 20 nm for each AZO layer and 30 nm of Al_2O_3. Using a scanning electron microscope, thorough and even coatings of each layer were observed. More tests are still needed in order to measure capacitance and capacitance density, but the preliminary results suggest that forming a capacitor on a high surface area AAO can produce devices with greater capacitance densities than what is currently being made with similar technology.

In Situ **Measurement of Stresses in Carburized Gears via Neutron Diffraction.** *Bryan Boggs* (***University of Tennessee at Martin, Martin, TN***); *Camden Hubbard* (***Oak Ridge National Laboratory, Oak Ridge, TN***). Carburized gears are characterized by a very hard outer layer that contains chemistry, phase, and microstructure gradients. X-rays have been used in the past to attain measurements of residual stresses, but X-ray diffraction is limited to near surface stress measurements due to attenuation. X-ray diffraction also has difficulty reaching the critical stress regions of a gear tooth due to beam interference from the complex geometry. This research seeks to develop experimental methods for measuring the stresses/strains in carburized gears at locations unattainable by X-rays and to do this as a function of applied load on the gear tooth. Experiments are being performed to determine if neutron diffraction can be used as an alternative to X-ray diffraction to measure the total stresses. Total stresses consist of both the residual stresses imparted during the carburization process and the load induced stresses resulting from power transmission. The experiments are being performed at the Neutron Residual Stress mapping Facility (NRSF2) at the High Flux Isotope Reactor (HFIR). In neutron diffraction, a powder sample is normally used to determine the unstressed lattice spacing or d_0. In carburized components, d_0 can not be determined from a single powdered sample because of the non-homogeneous material in the carburized region. As an alternative to using a powder sample, a method commonly used in X-ray diffraction known as the sin2σ method is being studied to determine if it can be used with neutron diffraction to accurately quantify d_0 in the carburized region. If successful, the sin2σ method will be used to determine d_0 at a number of points in the carburized region. Neutron diffraction methods will then be used to measure the d-space at each of the points for which d_0 was determined. The combination of d-space and d_0 at each point will enable the strains and stresses to be determined at the measurement points. The measurement of the d-space in a loaded gear is being facilitated with a Static Load Application Device (SLAD). This device was designed to statically load the gears as well as be compatible with the equipment at NRSF2. Stress analysis was done on the SLAD to ensure that the device would not exceed strength values found in engineering design standards.

Redox Reactions wiith Single-Walled Carbon Nanotubes. *Sofiane Boukhalfa* (***University of Illinois at Urbana-Champaign, Urbana, IL***); *Stephen Doorn* (***Los Alamos National Laboratory, Los Alamos, NM***). Single walled carbon nanotubes (SWNT) have been under close scrutiny by the scientific community since their discovery in 1992 due to their surprising material properties. SWNT synthesis can be achieved in numerous ways. However, these methods result in a wide spectrum of chiralities of nanotubes. In order to more efficiently use these novel materials, individual chiralities must be isolated.

To achieve this, a reduction-oxidation chemistry approach is used. Redox reagents of different electro-chemical potentials were added to solutions of SWNT in surfactant. Spectroscopy (absorbance and fluorescence) measurements were taken in order to monitor the effects of electron transfer between the SWNTs and the salts. Once chiral-specific doping can be achieved, isolation of these individual chiralities of SWNTs is planned through the use of ultracentrifugation. 20µL of each redox reagent was added to 3mL SWNT in surfactant solution while spectroscopy measurements were taken every 7 seconds in order to map out chiralities. Using a Fermi level map of the chiralities as a model, it was determined that the electro-chemical potential of the reagents directly affected the chiralities which were quenched during the experiments. Interaction between the salt and the surfactants is characterized by aggregation of the SWNTs; in the current redox reactions such aggregation, which broadens the spectroscopic signals, is not observed. Thus, a direct correlation was determined between the electro-chemical potential of the redox reagent and its effects on the nanotube solutions. This work establishes a future guideline for new work in the isolation of individual chiralities of nanotubes.

Spallation Neutron Source (SNS). *Yan-Jiun Chen* (*National Taiwan University, Taipei, Taiwan*); *Robert W. Shaw* (*Oak Ridge National Laboratory, Oak Ridge, TN*). The Spallation Neutron Source (SNS) uses a Multi-Turn Charge-Exchange Injection to form short pulses of protons in its accumulator ring. Carbon stripper foils are used in this process for removing electrons from the incoming H-Linac beam. Testing at various facilities has shown diamond stripper foils have an expected lifetime of more than 100 hours, five times that of evaporated carbon ones. Longer lifetimes can reduce beam downtime for replacement. This project involves producing diamond foils for the SNS and other accelerators. Diamond foils are grown via microwave plasma enhanced Chemical Vapor Deposition (CVD) on patterned silicon substrates. Substrates are patterned using photolithography, and etched chemically to produce corrugations 5~7 µm deep around the edges for mechanical stability of films. To enhance nucleation, the substrate surface is abraded with diamond powder slurry in an ultrasonic bath. Subsequently, 1~2 µm thick nano-crystalline diamond films are grown on the substrates under 1,000 W of microwave power and 130 torr with a gas mixture of 2% CH_4, 8% H_2, and 90% Ar. The substrate is then partially removed with a 1:1:1 mixture of hydrofluoric acid, acetic acid, and nitric acid, leaving a silicon edge for support of the foil. The main focus of the project is to optimize the performance of these diamond foils. Recently, foil areas have been increased by 57% to minimize beam loss, and foil thickness has been increased to maximize efficiency. Scanning electron microscopy (SEM) of the foils has revealed imperfections in the films, including black spots and pits. Black spots have not been proven to affect performance. Nevertheless, they can be successfully removed by etching away the surface of the film in a pure H_2 plasma. They are also suppressed with a lower growth pressure. Pits, ranging from 1 µm~10 µm wide, are suspected to cause loss of stripping efficiency and may result in holes or weak spots in foils. These pits are mainly caused by dust particles during the patterning of the substrates. Special attention is now paid to the preparation of substrates to minimize these occurrences. A cloudy visual appearance signifies uneven nucleation densities and/or substrate roughness. As a result, adjustments to the duration of ultrasonic abrasion are being made. Further testing in the SNS will reveal whether these measures lead to greater efficiency and longer lifetimes of foils.

X-ray Absorption Spectroscopy of Yb^{3+}-Doped Optical Fibers. *Robert Citron* (*The University of Chicago, Chicago, IL*); *Arthur J. Kropf* (*Argonne National Laboratory, Argonne, IL*). Optical fibers doped with Ytterbium-3+ have become increasingly common in fiber lasers and amplifiers. Yb-doped fibers provide the capability to produce high power and short pulses at specific wavelengths, resulting in highly effective gain media. However, little is known about the local structure, distribution, and chemical coordination of Yb^{3+} in the fibers. This information is necessary to improve the manufacturing process and optical qualities of the fibers. Five fibers doped with Yb^{3+} were studied using Extended X-ray Absorption Fine Structure (EXAFS) spectroscopy and X-ray Absorption Near Edge Spectroscopy (XANES), in addition to Yb^{3+} mapping. The Yb^{3+} distribution in each fiber core was mapped with 2D and 1D intensity scans, which measured X-ray fluorescence over the scan areas. Two of the five fibers examined showed highly irregular Yb^{3+} distributions in the core center. In four of the five fibers Yb^{3+} was detected outside of the given fiber core dimensions, suggesting possible Yb^{3+} diffusion from the core, manufacturing error, or both. X-ray absorption spectroscopy (XAS) analysis has so far proven inconclusive, but did show that the fibers had differing EXAFS spectra. The Yb^{3+} distribution mapping proved highly useful, but additional modeling and examination of fiber preforms must be conducted to improve XAS analysis, which has been shown to have great potential for the study of similar optical fibers.

Production of Pure Phase Multiferroic Materials by High Oxygen Pressure Annealing Processes *Matthew Cromwell* (*Brigham Young University – Idaho, Rexburg, ID*); *R.W. McCallum* (*Ames Laboratory, Ames, IA*). This study aims to form multiferroic materials from Rare Earth compounds and Manganese oxides. The desired composition is RMn_2O_5 (R = Rare Earth). Multiferroic materials import stems from simultaneous ferromagnetic and ferroelectric characteristics. Because of their dual nature, multiferroic materials could be utilized in various technologies. Understanding the properties and behavior of their crystal systems is critical to the eventual application of such materials, yet producing a pure single phase is still problematic. This must be done by reacting highly stable R_2O_3 with Mn-O, which remain stable in air to 1200°C. Existing phase diagrams for the given system indicate that increasing partial pressure of Oxygen (PO_2) greatly improves the kinetics of the phase transition. However, at such temperatures and pressure, standard materials soften and fail. Oxygen also presents a problem, being highly corrosive in nature. A furnace chamber maintaining up to 1000°C with 10 bar PO_2 was built from Hainesalloy-230, a high-nickel superalloy. This pressurized system was installed and various safety considerations and procedures were negotiated in bringing this into operation. A series of experiments varying annealing conditions were then performed to study the effect of changing PO_2. Two sets of samples were made from the same mixture and run at 900°C and 1000°C for twenty hours. Within each set, four pressures (in bar) were applied — 9.3, 3, 1 and 0.2. Results were examined by X-Ray Diffraction (XRD) to determine composition. XRD showed the content of desired phase increased significantly at higher temperature and also with increasing PO_2. Increasing temperature by 100°C produced double the desired phase over the 20 hour test. Still significant was the increase due to PO_2 which lifted the desired phase from 60% to 72%. This affirms the importance of PO_2 in this phase system. In further study, the effect of PO_2 over greater time intervals and elevated temperatures may be instructive in obtaining pure phase materials.

Zirconium Oxide Nanostructures Prepared by Anodic Oxidation. *Ying Yi Dang* (*Columbia University, New York, NY*); *M. Parans Paranthaman* (*Oak Ridge National Laboratory, Oak Ridge, TN*). Zirconium oxide is an advanced ceramic material highly useful for structural and electrical applications because of its high strength, fracture toughness, chemical and thermal stability, and biocompatibility. If highly-ordered porous zirconium oxide membranes can be successfully formed, this will expand its real-world applications, such as further enhancing solid-oxide fuel cell technology. Recent studies have achieved various morphologies of porous zirconium oxide via anodization, but they have yet to create a porous layer where nanoholes are formed in a highly ordered array. In this study, electrochemical methods were used for zirconium oxide synthesis due to its advantages over other kinds of coating techniques, and because the thickness and morphology of the ceramic films can be easily tuned by the electrochemical parameters, such as electrolyte solutions and processing conditions, such as pH, voltage, and duration. The effects of additional steps such as pre-annealing and post-annealing were also examined. Results demonstrate the formation of anodic porous zirconium oxide with diverse morphologies, such as sponge-like layers, porous arrays with nanoholes ranging from 40 to 75 nm, and nanotube layers. X-ray powder diffraction analysis indicates a cubic crystallographic structure in the zirconium oxide. It was noted that increased voltage improved the ability of the membrane to stay adhered to the zirconium substrate, whereas lower voltages caused a propensity for the oxide film to flake off. Further studies are needed to control and confine these morphologies to specific parameter windows, and to investigate other important characteristics such as ionic conductivity.

X-ray Absorption Fine-Structure Spectroscopy of a Platinum-Nickel Catalyst X-ray Absorption Fine-Structure Spectroscopy of a Platinum-Nickel Catalyst for a Direct Methanol Fuel Cell. *Jarod Delhotal* (*Sauk Valley Community College, Dixon, IL*); *Carlo Segre* (*Argonne National Laboratory, Argonne, IL*). Direct methanol fuel cell (DMFC) technology is approaching the point where it may solve many energy problems for portable power. However, there are still several obstacles that must be overcome before DMFCs become a viable energy solution. To better understand the structure of a platinum-nickel catalyst, researchers used X-ray absorption fine-structure (XAFS) spectroscopy to analyze the first-shell structure of a platinum-nickel cathode catalyst for a DMFC. The nanoparticle catalyst was supported by a carbon base and pressed into a proton-permeable membrane to form a membrane electrode assembly (MEA). The dry MEA was then scanned at both nickel K edge and platinum L3 edge energy ranges. Surprisingly, it was discovered that the nickel atom's most abundant near neighboring atom was oxygen at 2.07 Angstroms (Å), while a nickel neighbor was also found at 2.63 Å. From the platinum edge, a platinum neighbor was found at 2.69 Å and a nickel neighbor at 2.62 Å. Similar results were found for a wet MEA that was taken directly out of the DMFC. Nickel was found to have an oxygen neighbor at 2.04 Å and a nickel neighbor at 2.60 Å. The platinum edge spectrum for the wet MEA showed a platinum neighbor at 2.69 Å and a nickel neighbor at 2.63 Å. Further analysis is being conducted to determine the structure of the catalyst while it is in operation in a DMFC. A comparison of these findings will help scientists to understand the structural behavior of the catalyst while it is in use. This will lead to a better understanding of fuel cell catalysis, and could accelerate the development of fuel cell technology.

Data Processing and Analysis for the Superconductivity Program. *Michael Duitsman* (*University of Evansville, Evansville, IN*); *Victor Maroni* (*Argonne National Laboratory, Argonne, IL*). The Superconductivity for Electric Systems Program at Argonne is performing detailed characterization studies on $Y_1Ba_2Cu_3O_7$ (YBCO) superconducting films deposited on long-length metal-substrate tapes. One of the important research tools used in this research is Raman micro-spectroscopy. With Raman spectroscopy it is possible to determine phase composition and texture quality of the YBCO films. A large number of Raman spectra have been collected for this program. Each spectrum has to be processed to remove background noise, so that a representative Raman spectrum extrapolated to a horizontal baseline can be obtained for further analysis. Various types of computer programs are available to carry out these tasks. In this report the use of one such series of programs, the GRAMS series, is discussed. The work is focused on processing groups of spectra obtained from YBCO tapes for which the processing time and temperature were varied from end to end. The tape was then diagonally milled in intervals throughout its length, so that the effects of baking could be observed throughout the depth of the superconducting film. The samples were analyzed by applying the software for baselining, noise smoothing, spectral subtraction, and curve-fitting of such spectra. It is clear that baking, as well as the introduction of rare earth elements, has a significant effect on the performance of superconducting films.

Single Component Variation of Low Activity Tank Waste to Determine Chemical and Physical Behavior on Molten Ionic Salt. *Shaelah Easterday* (*Gonzaga University, Spokane, WA*); *Michael Schweiger* (*Pacific Northwest National Laboratory, Richland, WA*). Bulk vitrification is a process used to safely contain Low Activity Waste (LAW) on the Hanford Site into a vitreous waste form. This is done by heating the LAW mixed with glass-forming minerals in a large melting container lined with Castable Refractory Block (CRB). At temperatures above 275–325°C, sodium nitrate and sodium nitrite (NO_3+NO_2) in the LAW form Molten Ionic Salt (MIS) that penetrates the CRB. When this occurs, the MIS may be able to leach from the CRB into the environment. Feeds were prepared by drying a liquid LAW simulant with glass-forming minerals. The LAW composition was varied, one component at a time. Feeds were placed in silica crucibles of a similar porosity as the CRB, and heated to 500°C at 5°C/minute and held for 30 minutes. The feed was removed, and the crucibles were heated from 650°C to 1,000°C at 7°C/minute and held at 1,000°C for 60 minutes. To determine MIS penetration, the crucibles were weighed before and after each heat treatment. It was found that by replacing NO_3+NO_2 with any component but sulfate, MIS migration decreased. Replacing NO_3+NO_2 with sulfate had no effect on MIS migration. Acetate and chromate decreased MIS migration more than other components. This project provides important data for the Bulk Vitrification of LAW on the Hanford Site by allowing to asses the response of MIS migration in melter feeds to composition variations of the waste.

Degradation of Organic Light Emitting Diodes. *Eric Ellenoff* (*Stanford University, Stanford, CA*); *Joseph Berry* (*National Renewable Energy Laboratory, Golden, CO*). Organic light emitting diodes (OLEDs) currently degrade much faster than standard inorganic light emitting diodes. In order to improve OLEDs' lifetimes, it is necessary to measure their degradation in a controlled environment to determine what mechanism is responsible. A measurement setup was constructed to characterize the decay of an OLED's IV curve, emission intensity, and emission spectrum. A baseline for device performance and degradation was established by measuring their performance in a glovebox where they were surrounded by Argon with water and oxygen kept below 0.1 ppm. A second set of devices was tested outside of the glovebox in a prototype for a portable encapsulation device designed and built at NREL known as a "puck". By comparing the degradation of these devices to the set which was kept in the glovebox, we were able to determine how effective the puck was at protecting OLEDs. Over the course of a 165-hour test, the intensity of the OLED tested in the glovebox fell by 62% which indicates a half life of 115.5 hours. The relative emission of light at each wavelength remained constant throughout this test. The OLED which was tested in a puck degraded much more rapidly: it lost half of its intensity after every 19 hours of use, and degraded even when not in use. Furthermore, as the OLED became dimmer, its emission spectrum shifted, resulting in significantly less light being emitted between 540 nm and 660 nm. For these reasons, the puck proved to be inadequate for encapsulating devices. A second-generation test would feature the ability to leak controlled concentrations of reactants such as oxygen and water vapor to the device.

Synthesis of Microporous Materials Using Amino Triazoles. *Laura Engerer* (*Valparaiso University, Valparaiso, IN*); *John Schlueter* (*Argonne National Laboratory, Argonne, IL*). Microporous materials (MM) are commonly used in separation and storage of hydrogen fuel for energy applications. The objective of this project is to synthesize MM using zinc and copper ions and triazole ligands by a solvothermal process. Various combinations of Zn and Cu were mixed with 3-amino-1,2,4-triazole (AmTAZ) and 3,5-diamino-1,2,4-triazole (DamTAZ) and carbonate, oxalate, cyanate, thiocyanate, dicyanamide, and tricyanomethanide anions in a water/ethanol mix. They were heat treated to 80, 120, and 140°C with reaction times ranging from one day to two weeks. The materials were characterized by X-ray diffraction and gas adsorption techniques. From the X-ray powder diffraction patterns, most samples were crystalline in nature, whereas a few appeared to be amorphous. Based on the diffraction patterns, modifications were made to the original protocol aiming at eliminating multiple phases and to grow single-crystals. Analysis of the powder patterns provided a few trends. Copper ions were too easily reduced and therefore produce multiple phases in each experiment. This indicated that copper ions are undesirable for this particular system. Zinc has been shown to produce promising structures before this project and remains the most promising metal ion. Initially the DAmTAZ ligand did not show as much promise as its AmTAZ counterpart as none of the powders that were crystalline in nature contained DAmTAZ. Of the other anions incorporated into the framework the oxalate anion tended to combine with just the metal ion causing it to form crystals of the starting material. A few different combinations produced single crystals, but of these only two were appropriate for single crystal X-ray diffraction. The first structure was $K_2[Zn_5AmTAZ_4(Oxalate)_4]_{0.6}(H_2O)$. This structure is similar in contents but not structure to one of the materials previously synthesized in this project. The second is $[Zn_2(DamTAZ)_2(OH)(OH_2)]\ (NO)_3$. This second one is the first to be synthesized with the DAmTAZ ligand. Future inquiries should take pH into account and experiment with solvent size.

Cross-Sectional Polishing for Solar Cell Device Characterization. *Nathan Fast* (*California State University – Northridge, Northridge, CA*); *Bhushan Sopori* (*National Renewable Energy Laboratory, Golden, CO*). Fabrication of high efficiency solar cells requires a method to study the effects of new processes on the interactions and physical structure within a cell. An improved method for cross-sectional polishing of large areas of solar cells is described. This method produces a highly planar surface compared to the conventional way of preparing a sample by cleaving. The traditional method of cleaving or fracturing a sample works well for single crystalline wafers, however, creates undesirable surface morphologies for polycrystalline wafers or multi-layered devices such as finished solar cells. The discontinuities caused by the fracture mechanics of non-homogeneous samples create patterns that make it difficult to characterize the true nature of the materials under study. Solar cells cross-sectioned by this improved technique can be characterized by very high resolution electron-beam and optical imaging to measure the alloyed regions of front and back contacts, thickness of backsurface field, and other important physical properties of solar cells. In this method, a standard polycrystalline Si solar cell sample is prepared in a specially designed polishing chuck and secured with wax. The sample is sequentially mechanically polished by progressively decreasing the polishing grit size with chemical mechanical polishing (CMP) used as the last step. Optical microscope and scanning electron microscope (SEM) micrographs showing the planar results of this progressive polishing method are presented showing the improvement over the cleaving technique. This large area cross-sectioning permits statistically significant evaluation of many areas of the cell. The planarity of the sample edge makes it possible to perform a variety of atomic force microscope (AFM), conductive atomic force microscope

(CAFM), scanning Kelvin probe microscope (SKPM) and other scanning analyses over large areas in addition to more localized investigations such as with SEM.

Bending Kirkpatrick-Baez Mirrors for Neutron Focusing. *Anthony Fields, Jay Patel* (*South Carolina State University, Orangeburg, SC*); *Gene E. Ice* (*Oak Ridge National Laboratory, Oak Ridge, TN*). Neutron scattering is useful for analyzing the atomic structure and defect density of materials. Because neutrons have spin, an isotope-dependent scattering cross-section and are penetrating, neutron beams are particularly well suited for the study of magnetic materials, bulk materials and for the study of low Z or mixed Z structures. Several techniques for directing a beam of neutrons to a sample are available. While guide tubes and collimators work well for large sample sizes, focusing becomes increasingly important as the neutron probe dimension and sample sizes become small. The importance of high-performance nondispersive focusing optics for neutrons has just recently been recognized. In micro-focus experiments, we consider the need for convenient wavelength tuning and/or broad bandpass beams. This favors achromatic methods based on specular reflection. The Elliptical Kirkpatrick-Baez (KB) scheme offers the best flexibility and neutron gathering power, and can nondispersively image neutrons to small spots with high intensity and source-limited brilliance. The KB geometry uses crossed mirrors in grazing-incidence. With perfect elliptical KB mirrors, spherical aberration can be eliminated. We have adopted techniques for producing elliptical neutron mirrors by controlled bending. The need to control the slope errors in the mirrors is very important. The mirrors, the bending control mechanism, and supports are integrated as a unit. Both mirrors are attached to, and bent by a leaf spring mechanism. We have used a laser beam to simulate a thermal neutron beam for system calibrations; we determine the radius of curvature of the mirror as a function of bender settings (coupling force). Results show changes in the focal length (and subsequently in the radius of curvature) with micrometer setting. From these results we can determine the optimum radius of curvature of the mirrors for minimum slope errors. The mirror in the vertical plane sags under gravity and therefore, introduces a mixture of defocus and spherical aberration. We have designed passive corrections by a series of springs under the mirror. A computer program has been written to calculate the gravitational curvatures and slope errors as a function of the number of support springs and loading conditions. Simulation results show that the effect of one spring alone reduces the slope errors by a factor of 10 and with three spring supports, slope errors are reduced by over a factor of 100.

Blends of Thiophene-Based Dendrimers with Titania Nanoparticles for Use in Organic Photovoltaic Devices. *Talia Gershon* (*Massachusetts Institute of Technology, Cambridge, MA*); *David Ginley* (*National Renewable Energy Laboratory, Golden, CO*). Dendrimers (small branched organic molecules) offer an exciting alternative to typical semiconducting polymers in organic photovoltaic applications, as they are monodisperse and have virtually independently tunable optical, electronic, and architectural properties. Similarly, metal oxide nanoparticles offer a tunable, high-mobility alternative to the established organic electron acceptors. Since nanoparticles are many times larger than typical organic acceptors they may also help address issues in domain size limitations that appear in fullerene-based bulk heterojunctions. To explore these advantages a unique blend of the thiophene-based dendrimer, 4G1-3S, and anatase titania nanoparticles has been studied for use in organic photovoltaic devices. The charge transfer between these two materials was monitored as a function of solvent, drying conditions, and device architecture. Changes in film domain size and morphology were observed via SEM and AFM images. Devices were made under a variety of conditions via solution processing in air. Although no photocurrent was consistently extracted, several key conclusions were drawn regarding how these materials interact, which point toward the realization of a successful device.

Accelerated Wear Testing of Ultrananocrystalline Diamond-Coated Pump Seals. *Lindsey Goodman* (*Binghamton University, Binghamton, NY*); *Greg Krumdick* (*Argonne National Laboratory, Argonne, IL*). The performance of industrial chemical pumps can depend directly on the performance of the seals installed within the pumps. Using a chemical vapor deposition process developed at Argonne National Laboratory, scientists can coat standard pump seals with a thin layer of Ultrananocrystalline Diamond (UNCD™). Prior laboratory tests conducted at Argonne found that when subjected to identical conditions as uncoated seals, UNCD™-coated seals show increased energy efficiency and dramatically increased seal life expectancy. The objective of the experiment described in this paper was to design a system that can be used to verify these findings by subjecting UNCD™ coated and uncoated seals to accelerated wear conditions. The experimental protocol requires the pumping of an abrasive slurry of diatomaceous earth for 100 consecutive hours. Prior to their installation into the abrasive pump testing system, Raman spectroscopy and surface profilometry was used to analyze and characterize the surfaces of uncoated and UNCD™-coated seals. Before performing a complete 100 hour test with both identical pumps, the system was evaluated for robustness and durability using only the pump containing the uncoated seal. Critical temperatures, pressures, and flows were continuously recorded while abrasives were pumped for 100 hours. Upon completion of the test, components of the test system were inspected for damage due to the abrasives. Accelerated wear was found on system parts, requiring design modifications to the experimental system. Analysis was performed on the uncoated seal. Wear on this seal was evaluated by comparison between pre and post-experimental surface profile analyses. When system modifications are complete, both UNCD-coated and uncoated seals will be subjected to identical accelerated wear conditions. Wear on the seals will be compared using surface profilometry and Raman spectroscopy.

Optimization of Spray-Coated Organic Photovoltaics. *Renee Green* (*University of Pittsburgh, Pittsburgh, PA*); *Garry Rumbles* (*National Renewable Energy Laboratory, Golden, CO*). Organic photovoltaic devices, traditionally spin-coated, were made via an airbrush spray-deposition technique from a 1:1 poly(3-hexylthiophene) (P3HT) and [6,6]-phenyl-C61 butyric acid methyl ester (PCBM) blend in dilute (1 mg/ml each) solution. Devices were tested for dependence on solvent boiling point and annealing temperature. Working devices resulted from solutions prepared in chloroform, toluene, chlorobenzene, and para-xylene, with an initial maximum power conversion efficiency of over 2% (average 1.787%) from a p-xylene solution. Chlorobenzene was selected for use in further studies due to its small statistical spread of device efficiencies, high-quality smooth films, and comparable (1.15%) efficiency values. Annealing the device active layer at 120°C resulted in the highest power conversion efficiency among annealing temperatures ranging from 90°C to 200°C. Spray-coating is ideal for its ability to deposit highly dilute solutions, create multilayer organic devices, and expand the range of available analysis techniques by permitting the creation of high-quality thick films.

Development of an ASTM Graphite Oxidation Test Method. *Tyler Guldan* (*The University of Tennessee, Knoxville, TN*); *Timothy D. Burchell* (*Oak Ridge National Laboratory, Oak Ridge, TN*). Graphite, one of the three allotropes of carbon, is a very useful material because of its unique chemical structure and properties such as mechanical strength, chemical inertness, and electrical conductivity. In order to advance our knowledge of various graphite brands, further research must be conducted to gain a greater insight into the process and effects of oxidation on graphite properties. Although the key processes and controlling elements of graphite oxidation have been identified, the behavior of this material during and after oxidation is not well established. Knowledge of this behavior is crucial in understanding what happens to the various graphite components in nuclear reactors. Thermogravimetric analysis in a vertical furnace of large samples of NBG-18 graphite at the Oak Ridge National Laboratory (ORNL) has been used to characterize the oxidation resistance of this material, and to increase the scientific understanding of the relationship between the rate of oxidation and the flow rates of gases, temperature, and the intrinsic reactivity of graphite. This helps to identify the more oxidation resistant forms of graphite. In addition, comparative analysis of data collected on other graphite materials has been conducted, in order to identify a more expedient procedure for analysis of graphite oxidation data. The information gathered from these experiments and calculations is geared towards the development of an American Society for Testing and Materials (ASTM) test method for the oxidation of graphite. More research on all of the types of graphite is needed, but such results suggest that the current ORNL procedure using the vertical tube furnace may become a reliable ASTM test method.

Friction and Wear of Steel in Commercial Gear Oils. *Joel Hernandez* (*University of Puerto Rico, Mayagüez, PR*); *Robert A. Erck* (*Argonne National Laboratory, Argonne, IL*). Friction is one of the principal causes of lower efficiency of mechanical systems. Friction also produces wear, which has an impact of millions of dollars per year to the industry and transportation. To reduce the effect of wear and reduce friction, new lubricants for transportation systems, like motor and transmission oils and their additives, are being synthesized and tested. Using a CSEM ball-on-disk tribometer, the performance of a commercial gear oils was tested in a range of temperatures from 250°C to 1,000°C. For the test, a flat 8620 steel surface was slid against a 0.5 inch diameter type 52100 steel ball; a light bulb with an on-off controller was used to raise and control the temperature in the system. Over the course of five-hour tests, all the samples behaved different from oil to oil, some of them rising and lowering its friction coefficient (μ) with the changes of temperature, the same happened with wear. For two of the five oils, the μ at the start and the end was constant regardless the test temperature; for the other three oils the friction coefficient had no noticeable change with the rise in temperature. These three oils lowered its μ faster with proportionally with an increment in temperature. By using surface mapping microscopy, we analyzed the shape of the scars and measured the volume of material loss. It was found that the behavior of oils do not followed any concrete pattern with the rise of temperature; it can be caused by the formula use for the oil fabrication and the additives presents in them.

Slag Penetration in Coal Gasifier Refractories. *Brent Hicks* (*Brigham Young University, Provo, UT*); *S.K. Sundaram* (*Pacific Northwest National Laboratory, Richland, WA*). Coal gasification is a process used to convert coal in the presence of water, elevated temperature, high pressure, and a reducing atmosphere into high-value chemicals and fuels. A gasifier refractory lining protects the stainless steel shell of the gasifier from elevated temperatures and corrosive coal slag. Refractories composed primarily of Cr_2O_3 have been found the most resistant to slag corrosion, but they still fail to meet their targeted operational lifetime of three years. Experimental data on the slag-refractory interaction is necessary to develop models to 1) identify critical conditions at which refractory corrosion sharply increases 2) predict the service life of a gasifier refractory, and 3) discover processes/techniques to protect the refractories for extended life. Laboratory tests were conducted to determine the penetration depth of three slags representative of a wide variety of coals in the United States into five high-chrome refractories. The slags were pressed into pellets, placed inside a core-drilled refractory sample, and heat-treated in a controlled atmosphere furnace. Variables tested were refractory-slag combinations, different maximum temperatures and partial pressures of O_2, and thermal cycling between temperature extremes found in commercial gasifiers. Slag penetration depths were measured from spliced optical images of each refractory. Samples heated to 1,470°C for 2 hrs had average penetration depths ranging from 1.99±0.15 mm to approximately 27 mm; compare to 1.53±0.26 mm to 8.47±0.34 mm for samples heat-treated at 1,310°C for 2 hrs and 8.55±0.55 mm to approximately 26.2 mm for samples thermally cycled three times between 1,150°C and 1,550°C. AUREX 95P, a high-chrome refractory containing 3.3wt% P_2O_5, showed the least slag penetration of all studied conditions. P_2O_5 likely reacts with the slags to increase their viscosity and restrict molten slag penetration. Results indicate that lower temperatures correspond to less slag penetration; the slag is less fluid at 1,310°C than at 1,470°C. Scanning electron microscopy will be used to verify slag penetration depths for AUREX 95P because it was difficult to distinguish slag from P_2O_5 with optical microscopy.

Establishment of Manganese Oxide and Lanthanum Oxide as Atomic Layer Deposition Materials for Lanthanum Strontium Manganate (LSM) Electrodes. *David Honegger* (*Lewis & Clark College, Portland, OR*); *Jeffrey Elam* (*Argonne National Laboratory, Argonne, IL*). Nanostructured electrodes coated with thin LSM films show promise for increasing the efficiency of solid-oxide fuel cells by dramatically increasing the reactive surface area. Although the ability of atomic layer deposition (ALD) to conformably coat high-aspect-ratio substrates with monolayer control makes the process ideal for LSM deposition on these porous structures, ALD deposition of the individual materials that make up LSM, manganese oxide, lanthanum oxide and strontium oxide, is still in the pioneering phase. Moreover, the creation of LSM requires that the deposition conditions (e.g., substrate temperature) for the constituent materials be compatible. This study explored the deposition environments of manganese oxide and lanthanum oxide, and then found suitable conditions under which lanthanum manganate could be deposited. The results were confirmed with spectroscopic ellipsometry, X-ray fluorescence and an *in situ* quartz-crystal microbalance. Manganese oxide films were deposited on silicon in a substrate temperature range of 100°C to 300°C by using bi s(ethylcyclopentadienyl) manganese [$Mn(EtCp)_2$] and de-ionized water as reactants. Lanthanum oxide films were successfully deposited on silicon as well in a substrate temperature range of 175°C to 300°C by using tris(i-propylcyclopentadienyl) lanthanum [$La(IpCp)_3$] and ozone as reactants, creating a convenient temperature window of 175°C to 300°C in which to attempt lanthanum manganate deposition. Lanthanum manganate films were then deposited by alternating the exposure of the reactants between $La(IpCp)_3/O_3$ and $Mn(EtCp)_2/H_2O$. Film composition was controlled by altering the ratio of reactant exposures to the substrate. Successful growth of lanthanum manganate paves the way for LSM deposition via strontium-doping, and the eventual optimization of its electrical properties for use in solid oxide fuel cells.

The Optimization of Atomic Layer Deposition Parameters for Zinc Oxide and Aluminum-Doped Zinc Oxide Films as Transparent Conducting Oxides in Dye-Sensitized Solar Cells. *David Honegger* (*Lewis & Clark College, Portland, OR*); *Gregory Krumdick* (*Argonne National Laboratory, Argonne, IL*). Severely low efficiencies have outweighed the benefits of low-cost and the manufacturing ease of dye-sensitized solar cells (DSSCs). However, recent nano-scale developments are unlocking new possibilities and rekindling considerable interest in these solar cells. Novel high-aspect-ratio substrates are being fabricated to support more light-harvesting dye and create a more direct path for electrons in the DSSC photovoltaic circuit, but the enhanced efficiencies are still too low for widespread application. The deposition of thin transparent conducting oxide (TCO) films, such as zinc oxide (ZnO) and aluminum-doped zinc oxide (AZO), on the surface of these substrates can increase DSSC efficiency by providing a more efficient electron transport. Therefore, the effects of deposition parameters on the optical and electrical properties of thin TCO coatings on high-aspect-ratio substrates need to be studied in order to create an optimal film. This study measured the resistivities and optical transmittances of ZnO and AZO films on anodic aluminum oxide membranes using atomic layer deposition, a thin-film deposition technique involving a binary sequence of self-limiting surface reactions. Spectroscopic ellipsometry, spectrophotometry, and four-point probe resistance measurements were used to study the effects of varying oxidizer strengths (H_2O, H_2O_2, and O_3) and deposition temperatures (50–200°C). Results have confirmed that a weaker oxidizer such as H_2O, as well as a higher deposition temperature, creates a significantly more conductive film. Optical transmittance, however, trends in the opposite direction; stronger oxidizers such as O_3 and deposition at lower temperatures lead to more transparent films. These observations suggest that both deposition temperature and oxidizer strength affect the number of oxygen vacancies in n-type conductive ZnO films. Also, the 5% aluminum AZO films were observed to enhance the conductivity of ZnO without compromising a significant amount of optical transmittance. In conclusion, AZO films are seen as advantageous over ZnO films as TCOs, but the optimal choice of oxidizer and deposition temperature will depend upon the relative influences of conductivity and transparency on the overall efficiency of the DSSC. Further studies on the effects of annealing the films after deposition would be advantageous to a more complete understanding of the potential ZnO and AZO films have as TCOs in nanostructured DSSCs.

The Effect of Metal Deposition on the Electro-Optical Properties of Single Wall Carbon Nanotube Networks. *John Hunt* (*University of Tennessee, Knoxville, TN*); *Ilia Ivanov* (*Oak Ridge National Laboratory, Oak Ridge, TN*). Single wall carbon nanotube (SWNT) networks exhibit high flexibility and optical transparency which make them perfect candidates to replace brittle and expensive indium tin oxide (ITO) as electrodes in displays. However, SWNT networks do not have the excellent conductivity of individual carbon nanotubes due to the high resistance of nanotube bundles and nanotube-to-nanotube junctions. The focus of this research project was to explore the deposition of metals on SWNT networks as a means to improve their conductivity, without sacrificing transparency. SWNT networks were deposited on the surface of glass and varying amounts, ranging from 1 to 10 nm, of Au, Pt and Pd were deposited via physical vapor deposition. The optical absorbance and sheet conductivity of SWNT networks were measured for each metal loading. Small metal loadings (~1nm) were found to increase conductivity by approximately 50% with a 6% average increase in absorbance. Analysis of absorbance and conductivity measurements both indicated electronic interactions occurring between SWNTs and deposited metal. Temperature-resistance measurements were used to determine the nature of this interaction, which was shown to be the reduction of barrier hopping resistance at nanotube-nanotube junctions.

Atomic Layer Deposition of Alumina on High Surface Area Silica Powders. *Trang Huynh* (*DePaul University, Chicago, IL*); *Jeffrey Elam* (*Argonne National Laboratory, Argonne, IL*). Alumina is a transparent insulating oxide which is widely used as a catalyst support layer. Thin films of alumina can be grown by atomic layer deposition (ALD) down to the nanoscale of one monolayer per reaction cycle by using trimethylaluminum and water. The purpose of this project was to determine whether nanoporous silica powders with high surface areas could be uniformly coated with alumina by the ALD method such that full saturation could occur. X-ray fluorescence and direct weight measurements were used to determine the growth rate of alumina with respect to the number of reaction cycles during growth. In order to better understand the surface chemistry of these powders, the properties of the alumina coated powders were analyzed using scanning electron microscopy and energy dispersive analysis of X-rays (EDAX) measurements. These measurements revealed uniform infiltration of the high surface area powder by the ALD alumina coating. There is future interest in growing cobalt oxide on silica powders by ALD for its potential use as a catalyst in nanoporous membranes using alumina as a support.

***In-Situ* Stress Measurement for MOCVD Growth of High Efficiency Lattice-Mismatched Solar Cells.** *Alejandro Levander* (*Pennsylvania State University, State College, PA*); *John Geisz* (*National Renewable Energy Laboratory, Golden, CO*). Dislocations, formed in order to relieve stress, act as sites for nonradiative electron/hole pair recombination, which reduces the efficiency of photovoltaics. Stress forms as a result of mechanical and thermal mechanisms during the metal-organic chemical vapor deposition growth process. Mechanical stress is the result of depositing lattice-mismatched (LMM) layers on top of one another and thermal stress results from a thermal gradient within the sample and depositing layers with different thermal expansion coefficients on top of one another. To reduce the number of dislocations in the active layer when using LMM materials, compositionally step-graded layers and a buffer layer are placed between the two LMM materials. In order to achieve a better understanding of the effect of stress on the active layer, the buffer composition, and therefore lattice constant, was varied to change the stress on the active layer. The *in-situ* stress and *ex-situ* strain were characterized using a multi-beam optical stress sensor and X-ray diffractometry respectively. The quality of the photovoltaic devices was measured using a solar simulator and quantum efficiency instrument. Samples with near zero stress or small amounts of compressive stress in the active layer had the highest open-circuit voltages and efficiencies. Tensile stress in the active layer significantly degraded performance. The biaxial modulus was calculated from a stress v. strain curve, but several sources of error exist. The band gap of the active layer increased with increasing stress, despite the composition remaining constant. Future work will concentrate on the effect of dopant type on stress development and dislocation formation in the graded layer.

Quasielectrostatic Carbon Orientation for Lithium-Ion Battery Applications. *Cliff McCold* (*Vanderbilt University, Nashville, TN*); *Jane Y. Howe* (*Oak Ridge National Laboratory, Oak Ridge, TN*). In Quasielectrostatic Carbon Orientation (QCO) processing, carbon precursor materials are thermally treated while subject to an alternating current (AC) electric field with the goal of producing carbon materials with oriented graphene sheets. To provide proof-of-principle, QCO-treated samples are characterized to determine if conductive graphene sheets composed of bonded carbon atoms can be aligned in the direction of the electric field. Carbons with oriented graphene sheets could potentially be used in lithium-ion battery anodes, permitting higher charge rates, greater maximum current, and higher power density. A tube furnace was used to raise the temperature of the precursor material in an inert argon atmosphere. The material was held in a rig between top and bottom capacitor plates at different voltages, which applied the field during the entire 24-hour thermal processing. Variables included precursor materials (Mitsubishi AR mesophase pitch, phenolic resin), highest temperature (280°C, 650°C), AC field frequency (200 Hz, 10 kHz, 700 kHz), and electric field strength (no field, 2.4 kV/cm, 6.0 kV/cm). Created samples underwent X-ray diffraction and impedance spectrometry to determine if desired graphene sheet orientation was achieved. Results indicated that AR mesophase pitch does not respond to QCO processing at softening temperature (280°C). Severe foaming of AR mesophase pitch under numerous run conditions up to carbonization temperature (650°C) excluded carbonized samples from both characterization techniques. Phenolic resin samples showed QCO treatment-dependant results at carbonization temperature, but data was from too small a sample population to see clearly defined trends. Future work includes extensive sample creation under various QCO field frequencies. The ability to apply a stronger field (6.0 kV/cm and higher) was only recently developed by minimizing capacitor plate separation, and many samples remain to be created using stronger field conditions. Isotropic pitch and cellulose are potential future precursor materials. Created samples must be analyzed using the X-ray diffraction and impedance spectrometry, and scanning electron microscopy will also be used for future analysis. Once a clear trend in data is observed, the appropriate precursor material and run conditions will be used to create samples for direct lithium-ion battery anode testing.

Safety Barrier Program to Protect Experimenters in the Event of Pressure Cell Explosion. *Rachel Morris* (*Maryville College, Maryville, TN*); *Lakeisha Walker, Louis Santodonato* (*Oak Ridge National Laboratory, Oak Ridge, TN*). In neutron scattering research, high pressure experimentation with pressure cells is becoming more prevalent. Although this particular type of experimentation can provide a plethora of information regarding solutions to geophysical issues as well as hydrogen storage designs, material analysis at high pressure can be very dangerous. For this reason, scientists need to take precaution for ensuring their safety when using pressure cells under extreme conditions. Since pressure cell fragments can be projected if the cell ruptures or explodes, a safety barrier should be constructed to contain the hazard. Therefore, I have written a program in Visual Basic for Applications using a Microsoft Excel spreadsheet interface to help determine an appropriate and cost-efficient safety barrier. The logic of the program is not only based on calculations derived from the equations of motion in physics for radial distance and velocity, but it is also based on equations of acceleration involving the thickness and density of the cell initially and the amount of pressure used in the experiment. These calculations are the basis for determining how fast an infinitesimal fragment (in the worst case, the entire cell) will be traveling at any given distance from the initial starting position of the cell. The primary purpose of the program is to use a model of velocity versus distance to aid in determining the dimensions for a safety barrier that will be impenetrable for the largest fragment traveling at the highest velocity. Although any cell can be modeled with this program (provided the user supplies certain values for variables), the Titanium Zirconium (TiZr) alloy cell is exampled in this paper, which has a pressure equivalent to 200×10^6 Pa, a density of 5,230 kg/m^3, a thickness of 0.0039751 m, and an initial radius of 0.003175 m. The TiZr cell was modeled for 35 steps for 0.000036 s, until the radial distance of the fragment reached 0.123 m and the velocity reached 195 m/s. Thus, the projected and desired results were achieved using the program, and the construction of the barrier will occur in fall 2007.

Model Based Illumination Optimization for a 003-Numerical Aperture Extreme Ultraviolet Lithography Tool. *Jonathan Nation* (*University of Arizona – Tucson, Tucson, AZ*); *Patrick Naulleau* (*Lawrence Berkeley National Laboratory, Berkley, CA*). There have been many recent advances in high numerical aperture (NA) extreme ultraviolet (EUV) lithography systems. The 0.3-NA Micro Exposure Tool (MET) at the Advanced Light Source (ALS) currently utilizes a programmable coherence illuminator system with the capability of creating different pupil fills. It is well known that illumination settings can be tailored to optimize printing performance for particular features. The optimal illumination settings, however, depend not only on the feature type but also on the specifics of the pupil function, including phase (or aberrations) and amplitude (or pupil obscurations). In order to maximize the productivity of the MET, the best possible pupil fill should be chosen for the feature type being imaged in each experiment. In this research, aerial image modeling software is used to study the optimal illumination conditions for the SEMATECH Berkeley MET tool as a function of feature size and type taking into consideration the known pupil function of the optic. The metrics of maximum contrast over a 50 nm focus range and depth of focus with contrast greater than 0.5 are used to gauge the performance of each pupil fill tested. We will present the best annular, dipole, and monopole pupil fills for each feature type as well as for each individual feature size. Experiments relying on only one feature size will obtain the greatest improvement in contrast and depth of focus by using the presented pupil fills, at the possible expense of losing quality of other feature sizes. The results are also directly compared to the default annular 0.35–0.55 setting used in the commercial implementations of the MET tool. Initial results for annular pupil fills reveal that improvements of up to 12% in each metric can be obtained.

Terahertz Spectroscopy of Chalcogenide Glasses. *Jeffrey Neumann* (*University of Michigan, Ann Arbor, MI*); *S.K. Sundaram* (*Pacific Northwest National Laboratory, Richland, WA*). Chalcogenide glasses are well known for having technologically significant optical properties such as high transparency in the infrared regime (some being transparent in the visible region), non-linear optical properties, semiconducting properties, and photomodifiable properties which make them attractive for applications in chemical and remote sensing. However, their properties in the terahertz (THz) regime are not reported in the literature. With the recent advances in the area of THz imaging and spectroscopy, advanced tools are now available to characterize this family of materials. Selected chalcogenide glasses were characterized using a THz spectrometer that covers a frequency range of 172–265 GHz and the data is presented here. Using a terahertz wave generator, terahertz waves were transmitted through a sample and then detected on the other side. Each sample produced a unique output displaying the magnitude of transmission across the frequency range. Software was used to fit the Fresnel expressions for complex transmission to the resulting graphs to determine the transmission, refractive index, and dielectric constants of each sample. The results showed that transmission of terahertz waves show a marked decrease as the waves move up into a higher frequency range. This decline is also varied in connection with the refractive index and whether it was increasing or decreasing.

Heat-Reflective Paint for Deck Surfaces of Naval Vessels. *Emily Ottenweller* (*University of St. Francis, Fort Wayne, IN*); *Rick Lowden* (*Oak Ridge National Laboratory, Oak Ridge, TN*). The new V-22 Osprey aircraft has the capability to vertically take off from and land on the deck of an aircraft carrier. During take off and landing, the hot exhaust from the Osprey aircraft engines impinges directly onto the deck of the ship. The deck surfaces were not designed to handle high temperatures and thus warp from the excess heat. Insulating paints that use special ceramic additives have been developed to improve the energy efficiency of buildings and structures. The decks of naval vessels are coated with paints that include ceramic grit to make them non-skid. It was hypothesized that the insulating additives could be used to replace the non-skid grit and create a paint that could protect the deck from the hot exhaust. Ceramic particles were added to an epoxy-based surface coating to alter the layer's thermal properties, i.e. to reflect, absorb or conduct heat. The ceramic additives include Bionic Bubbles which are hollow microspheres derived from fly ash, Insuladd particles which are hollow ceramic mircospheres invented by NASA, and silicon carbide platelets. The test specimens were ½ inch thick steel plates to which paints with different additives were applied. The plates were heated using a heat gun to simulate the exhaust of the aircraft and temperature distributions on the backside of the painted specimens were measured. The temperature distributions for the plates coated using paints with additives were compared to results for bare metal, paint with no additive, and the standard non-skid deck coating. The plate with the highest concentration of Bionic Bubbles was the most effective in reducing the temperature of the plate; however, the layer is likely too fragile for the application. The deck coating must not only be insulating but also robust and non-skid. It appears that the ceramic additives will need to be modified to best suit the needs of this application.

Developing Minimally Resistive and Highly Transparent Conduction Oxides Films by Atomic Layer Deposition. *Victor Oyeyemi* (*Goshen College, Goshen, IN*); *Jeffrey Elam* (*Argonne National Laboratory, Argonne, IL*). Zinc oxide (ZnO) and indium oxide (In_2O_3) are important members of the group of oxides known as transparent conducting oxides (TCOs), which are used in thin film coatings for such applications as energy-conserving windows, surface electrodes for flat panel displays and solar panels, and invisible security shields in windows. The electrical and optical properties of these oxides depend on the method of preparation and can also be modified by the incorporation of dopant materials. ZnO and In_2O_3 films can be prepared in various ways including spray pyrolysis, sputtering, chemical vapor deposition and pulsed laser deposition. Researchers at Argonne National Laboratory are investigating a new technology known as atomic layer deposition (ALD), which uses the self-limiting surface reactions of the precursors (reactants), sequentially applied to the surface, to generate thin films one molecular layer at a time. Thus, film thickness can be controlled with precision on an Angstrom level. In particular, ALD is more advantageous to traditional methods because it produces films that are smooth and conformal. This report presents a study to develop TCO films that are both more conductive and transparent than what is currently available. There are two parts to the study. The first part involves establishing the growth rate, morphology, and crystalline structure of ZnO films produced by the ALD of diethyl zinc and ozone precursors, and how these properties are affected by the growth temperature. The effect of aluminum doping on the ZnO film resistivity was also examined. The second part of the study examined the effect of zinc doping on the resistivity, transparency, and crystalline structure of ALD grown In_2O_3 films, as well as how these properties depend on growth temperature. The pure zinc oxide films deposited at 150°C were shown to have a linear growth rate of ~0.46Å/cycle and a resistivity of 0.26–0.6 Ocm. Contrary to expectations, the aluminum-doped ZnO films deposited at 200°C were found to have a strictly increasing resistivity with respect to doping level. Investigation remains ongoing to obtain a complete picture of the properties outlined above.

Control of Carbon Nanofiber Alignment During Growth in Plasma Enhanced Chemical Vapor Deposition Processes. *Ryan Pearce* (*University of Tennessee, Knoxville, TN*); *Michael Simpson* (*Oak Ridge National Laboratory, Oak Ridge, TN*). Carbon nanofibers are just recently coming under scrutiny with a number of potential uses such as gene delivery arrays and neuronal interfaces. Nanonofibers have a stacked "herringbone" structure, giving them a very high aspect ratio, which is what lends them such a great range of possible applications. Carbon nanofibers are typically grown using a process called "Plasma Enhanced Chemical Vapor Deposition" (PECVD). In this process, a silicon wafer with nickel deposited on it in a specific pattern is placed on a heater in a vacuum chamber. The heat is turned on and ammonia (NH_3) and Acetylene (C_2H_2) are introduced into the chamber at a specific pressure and flow rate. A plasma is induced which causes the carbon from the C_2H_2 to deposit on the nickel dots, forming the carbon nanofibers. This process forms vertically aligned carbon nanofibers. Our project aims to find a technique to control the alignment of the nanofibers during growth. The way we do this is by changing the method whereby gas is introduced into the system. Classically, the gas flows into the chamber perpendicular to and far away from the surface of the wafer, so that only the ratio of gases affects the growth, and not the flow rate. What we do is place a nozzle directly over the wafer so that the gas flows directly over it. We devise an optimal regimen for growth where we only vary the total flow, keeping the ratio of gases constant. After growth, we observe the wafer under a scanning electron microscope. We have found that nanofibers respond to the variations in flow by tilting along the flow. There are some aspects of this study that require further investigation. First, we need to determine the correlation between angle of tilt and flow rate, which can be done by setting up a series of experiments keeping the total flow variable in a stepped sequence and measuring the resulting angle formed by the nanofibers and the substrate. Then, flow ratio needs to be varied to determine the resulting effects. Another method of creating tilt in nanofibers is through variance in the electric field during PECVD. Towards the edge of the field, this causes the nanofibers to "bend." This technique is impractical, however, as only the fibers grown on the very edges of the field are affected. Our study is fundamental in understanding how to control nanofiber growth, which will lead to an overall better understanding of nanoscale fabrication.

Self-Assembly of Au Nanorods. *Eric Petersen* (*Harvard University, Cambridge, MA*); *Jin Wang* (*Argonne National Laboratory, Argonne, IL*). The packing of Au nanorods into ordered arrays was studied. A seed-mediated growth process was used to synthesize Au nanorods in surfactant with lengths of approximately 30 nm (aspect ratio ~4). UV-visible spectophotometry and transmission electron microscopy were used to characterize the nanorod dimensions. The behavior of the nanorods under an applied dc electric field of 5,000 V/m was monitored *in situ* by small angle X-ray scattering and two-photon photoluminescence microscopy. Scanning electron microscopy was used to observe the nanorod arrays left behind on the electrode surface after the field was turned off. Results indicate that a net positive charge remains on the Au nanorods after synthesis. This charge encourages the formation of two types of ordered nanorod domains; one with the longitudinal dimension oriented along the field lines, the other with it oriented normal to the field lines. Future research will investigate charge properties of Au nanorods in organic solution, and on different types of electrodes, with the objective of understanding their electrical behavior. This understanding may be applied to create large scale domains of Au nanorods with a single orientation, for a wide array of device applications.

Compiling and Organizing RIMS-Related Data. *Walter Pettus* (*Hillsdale College, Hillsdale, MI*); *Michael R. Savina* (*Argonne National Laboratory, Argonne, IL*). Investigation of microscopic silicon carbide grains found in meteorites has revealed isotopic ratios which closely match the predicted values based on the nucleosynthesis models, but are unlike anywhere in the solar system. This has led scientists to classify the grains as presolar, having been unchanged since their creation in stars before the formation of our solar system. Using Resonant Ionization Mass Spectrometry (RIMS) at Argonne, investigations into heavier elements have been made possible. The existing data associated with this project was scattered throughout many published articles and several electronic files. In order to expedite the processes of research and analysis, it was necessary to organize all the available data into searchable databases. The first phase was to gather all the RIMS data along with corresponding standards and laser information and to sort it into a Microsoft Access database. The second phase was to organize the data collected regarding the Titanium:Sapphire laser cavity configurations so that when tuning the lasers back to the same wavelength in the future, an optic arrangement could be quickly recreated to optimize power. Finally the RIMS data was reformatted and exported so that it could be combined with the online database of all presolar grain data that Washington University in St. Louis is constructing. The databases that have been created hold the formatting so that all future data obtained can be easily entered and the databases maintained with all the available data.

Cd_2SnO_4 and ZnMgO: Ternary Transparent Conducting Oxides for Thin Film CdTe and $CuInGaSe_2$ Solar Cells. *Hannah Ray* (*Wesleyan University, Middletown, CT*); *Xiaonan Li* (*National Renewable Energy Laboratory, Golden, CO*). The compounds cadmium stannate (Cd_2SnO_4) and zinc magnesium oxide alloy ($Zn_{1-x}Mg_xO$) are ternary transparent conducting oxides (TCOs) that are promising for use in thin film solar cells. When used in place of the commonly used TCO tin oxide (SnO_2), Cd_2SnO_4 enhances small-scale cadmium telluride (CdTe) solar cell performance. To scale up the deposition area and rate of Cd_2SnO_4, metal organic chemical vapor deposition is used. The commonly used precursors for cadmium oxide and SnO_2 have incompatible deposition temperatures; thus, a different tin oxide precursor with a lower deposition temperature must be found to be used with dimethyl cadmium. A literature search was performed, and tin (II) acetylacetonate was deemed best for this application. Zinc oxide (ZnO) is commonly used in copper indium gallium diselenide (CIGS) solar cells. Alloying ZnO with the insulator magnesium oxide (MgO) allows for band gap (Eg) engineering. However, adding MgO to a ZnO alloy detracts from the film's electrical conductivity. In this study, a gradient of ZnO:Al and MgO was deposited using RF magnetron sputtering by two ZnO:Al and MgO targets, creating the zinc magnesium oxide alloy $Zn_{1-x}Mg_xO$. Magnesium (Mg) content of the films varied from 3–30%. The optical and electronic properties of the film were measured every centimeter along the gradient. It was determined that the electrical properties of the $Zn_{1-x}Mg_xO$ alloys are only acceptable for use in a solar cell when the proportion of Mg in the sample is less than 3.5% (xg ($Zn_{1-x}Mg_xO$) = 1.72x + Eg (ZnO) with the amount of Mg in the alloy. At Mg content between 0–7%, the band gap was higher than this fit line due to the Burstein-Moss effect. As Mg content increased in this region, Eg due to the Burstein-Moss effect decreased until the effect disappeared at x = 0.07. Increasing the deposition temperature improved the electronic conductivity of the films. Increasing the temperature had no effect on the band gap when the Mg content was greater than 7%, but when Mg content was less than 7%, Eg increased with temperature.

Synthesis and Integration of Carbon Nanofibers for Nanobiological Applications. *Patricia Reynolds* (*University of Toledo, Toledo, OH*); *Michael L. Simpson* (*Oak Ridge National Laboratory, Oak Ridge, TN*). Recent advances in the controlled synthesis of nanomaterials are enabling new approaches for probing biological system functionality at the nanoscale. Several CNMS projects incorporate one such nanomaterial, carbon nanofibers, as functional elements. Applications include the use of carbon nanofibers as nanoelectrodes, as means for gene delivery, and as the principle component of biomimetic membranes. Current efforts make use of vertically aligned carbon nanofibers (VACNF) as suspension supports for lipid bilayers (LBL). There are two main purposes; one is to develop techniques to form LBL structures and secondly to use them as a platform to study integral membrane proteins and their interactions using neutron scattering. Carbon nanofibers are synthesized in a plasma enhanced chemical vapor deposition process from metal (nickel) catalyst, and the vertical alignment results from the applied electric field during synthesis. The growth process allows for controlled synthesis as the parameters (e.g., length, diameter, shape, position, and chemical composition) of individual nanofibers can be selected by definition of catalyst properties and growth conditions. LBLs are composed of phospholipid head groups which are hydrophilic and hydrocarbon centers which are hydrophobic. To fabricate hybrid structures composed of VACNF arrays with deposited LBLs, small unilamellar lipid vesicles were generated through the sonication of LBLs, deposited on VACNF arrays, and heated to 35 degrees C for 30 minutes to encourage layer development. Atomic Force Microscopy was used to map the VACNF array surface both before and after LBL deposition, and encouraging initial results were acquired to show a change in surface topography that is indicative of the formation of a LBL on the nanofibers. The next step in this work is to image these hybrid structures with neutron scattering to validate the presence of a suspended LBL on the surface of carbon nanofibers.

Elaboration on the Hexagonal Grid and Spiral Method for Data Collection via Pole Figures. *Anthony Rizzie* (*Ball State University, Muncie, IN*); *Thomas Watkins* (*Oak Ridge National Laboratory, Oak Ridge, TN*). A pole figure provides a representation of the distribution of a particular set of atomic planes for data acquired through diffraction and is used for analyzing crystallographic texture or preferred orientation. Pole figures are constructed from a collection of data points, each with a prescribed azimuthal angle (phi) and sample tilt angle (chi) (as specified by the goniometer) and measured intensity (counts or counts per second). Traditionally, the Schulz method (5°x5° grid) is employed to acquire the necessary data, but this leads to a high concentration of data points for small chi values, low concentration for large chi values, and consequently an inefficient use of time. Two alternative data collection methods, the hexagonal grid and spiral, have been previously proposed but only tersely documented in terms of both construction and implementation. The goal, therefore, is to provide a practical description of the mathematics required to implement the hexagonal and spiral data collection schemes. Applying the concepts of equal area and stereographic projections and geometry, spreadsheets were created to formulaically develop hexagonal and spiral grids, which are then related to angular movements of the goniometer. Using the generated data points, the hexagonal grid and spiral methods were programmed by "brute force" into the existing X-ray software and employed to collect data for a sample of aluminum foil. The resulting (111) pole figures compared favorably to typical rolling textures for aluminum foil collected with the conventional Schulz method. The hexagonal grid has been shown to reduce the number of data points and time needed to complete a pole figure, while providing equal area sampling. The spiral method was shown to use only a quarter as many data points as the 5° x 5° grid. In the future, LabVIEW software will be utilized to develop programs for collecting data using both the hexagonal grid and spiral methods and then convert the data back to the conventional 5° x 5° grid.

Synthesis of leucite nanocrystals through glass hydration and sol-gel method. *Elisa Sanchez* (*San Antonio Community College, San Antonio, TX*); *Pavel Hrma* (*Pacific Northwest National Laboratory, Richland, WA*). Leucite ($KAlSi_2O_6$), a potassium alumino-silicate, has been widely used as a principal component in porcelain-fused-to-metal restorations to match the thermal expansion coefficient (CTE) of dental porcelain with the CTE of the metal. The combination of porcelain and metal creates a strong and durable restoration that is highly biocompatible and wear-resistant but lacks the natural translucency of enamel. The low strength and fracture toughness of natural looking all-ceramic restorations have restricted their widespread use in aesthetic dentistry. The introduction of nanoleucite porcelain might make the all-ceramic restorations possible. It was found that the fracture toughness and flexural strength of the leucite porcelain can be significantly increased by controlling the amount, particle size, and distribution of the leucite crystals in the porcelain matrix. The objective of this study was to synthesize nanocrystals of leucite via glass hydration and sol-gel method where the silica sol was mixed either with potassium aluminate for a basic synthesis or with potassium nitrate and aluminum nitrate for an acidic synthesis. The synthesized powders were analyzed with X-ray diffraction (XRD) to determine the concentration of leucite, and with scanning electron microscopy and energy dispersive spectroscopy (SEM-EDS) to determine the crystal size of leucite. Differential thermal analyses and thermogravimetric analyses were used to study the phase changes in amorphous gels leading to the formation of leucite. Preliminary results of XRD analyses of the glass hydration samples show crystal formation. The concentration of leucite increases with temperature, time, and added volume of water. Average crystal size as determined by SEM was approximately 1 µm. At the time of this report, sol-gel data collection is ongoing, but nearing completion. Preliminary results of this study suggest a promising trend toward synthesis of nano-sized particles of leucite using both tested methods.

Uniform Height, Self-Organized Pb/In Islands Grown on Si(111) 7 x 7 Interfaces. *George Scott* (*Northwestern University, Evanston, IL*); *Michael Tringides* (*Ames Laboratory, Ames, IA*). The ability to grow uniform height self-organized nanostructures is made possible by Quantum Size Effects (QSE) and could have applications in the technology sector. Pb deposited epitaxially onto different Si(111) interfaces ((7 x 7), Beta Phase-sqrt(3)xsqrt(3), Alpha Phase-sqrt(3)xsqrt(3) etc) produced islands of uniform heights but In on Si(111) 7 x 7 did not. These initial Si(111) interfaces are prepared by depositing small amounts of Pb or In under ultra-high vacuum (UHV) conditions. After these initial 2-D phases are prepared, deposition is followed by the element of interest (Pb or In) to form epitaxial nanostructure islands. Good height uniformity was found when In was deposited on the Pb Alpha Phase-sqrt(3)xsqrt(3) phase when fcc In(111) islands are present. In order to gain insight into the kinetics that could lead to similar behavior in other Pb-In mixed systems, Pb was deposited on In sqrt(31)xsqrt(31). Spot profile analysis — low-energy electron diffraction (SPA-LEED) was the primary method used in the uniform height experiments because of the ability to observe a large sample area. From the diffraction intensity profiles, uniform heights existed only when Pb was present in the system. Scanning tunneling microscopy (STM) was also used to build height histograms and verify the diffraction results.

Combinatorial Study of $Zn_x Sn_{1-x} O_y$: a Transparent Conducting Oxide. *Diana Silva (University of Colorado, Boulder, CO); Maikel Van Hest (National Renewable Energy Laboratory, Golden, CO).* Zinc-tin-oxide (ZTO) libraries were co-sputtered from ZnO and SnO_2 by RF magnetron sputtering onto Eagle 2000 glass substrates at temperatures ranging from room temperature to 550°C and analyzed with combinatorial analytical techniques. Samples were deposited in pure argon and in a mixture of argon and hydrogen with 4% and 2% hydrogen. ZTO films were found to be amorphous at a tin content ranging from 30 to 70% when deposited in pure argon gas at 550°C. ZTO films were also amorphous when deposited in a mixture of argon/hydrogen with 4% and 2% hydrogen at 550°C. ZTO films deposited in pure argon had a maximum conductivity of 134 S/cm at a 70% tin content and an optical transparency with transmission 85%. While, ZTO films deposited in a mixture of argon and hydrogen 2% had an optical transparency with transmission 85% across the visible spectrum and a maximum conductivity of only 92 S/cm observed at 75 at % tin content. Thin films of zinc-tin-oxide deposited in a mixture of argon and hydrogen with 4% and 2% hydrogen, did not improve conductivity and optical properties as expected. Key words: zinc tin oxide, transparent conducting oxides, amorphous.

The Effects of Polymers on the Rheology and Green Strength of the Feedstock in Titanium Injection Molding (TIM). *Juan Teran (Estrella Mountain Community College, Avondale, AZ); Kevin L. Simmons (Pacific Northwest National Laboratory, Richland, WA).* When implementing naphthalene as a patented primary ingredient in our binder system, it is imperative that we understand and assess the characteristic differences that polymers have on the torque values and green strength of the overall mixing with titanium hydride powder. Knowing how to influence the torque values during the initial mixing between the binder and powder will give us a better chance of molding a complex geometrical shape, since increasing the powder volume is known to increase the torque value and decrease the mold capacity of the mix. By keeping the volume for all binder constituents' equal, and changing the chemistry for one of the constituents, in this case acetamide, low density polyethylene, and polyethylene glycol, the results of these experiments can provide insight on the effects that these alternative constituents have on the feedstock's rheological properties as well as its green strength. Each individual run was carefully mixed and monitored in a Haake Rheocord Fisions 90 torque rheometer and their torque values were recorded in Newton-meters. All mixes were conducted in the same manner and were allowed to reach their lowest torque value within a forty minute time limit. Acetamide, when in mixture with stearic acid and naphthalene, produces the lowest torque and injection molds but does not have enough green strength to be handled and sintered. The polymer mixtures produced different torque values that were slightly higher than that of acetamides but can be injection molded nicely with suitable green strength to pass through the sintering stage. This work was focused on analyzing the effects that specific polymers have on the feedstock traits, in anticipation of finding one adequate enough to increase its probability of entering the fourth stage in the TIM process. The use of polymers in the naptha-based binder is necessary; they provide key properties to the feedstock that allow it to pass through the entire TIM operation. Ongoing research and development continues in the areas of binder development, molding conditions, and proper heat treatment during the de-binding and sintering phase to improve the overall success rate in the production of viable parts in the TIM process.

The Effect of MnO on the Thermal, Mechanical and Interfacial Reaction of a SOFC Sealing Glass with Metallic Interconnect Materials. *Jameson Thornton (Santa Rosa Junior College, Santa Rosa, CA); Yeong-Shoung Chou (Pacific Northwest National Laboratory, Richland, WA).* This paper will discuss the effect of MnO on the thermal and mechanical properties of a novel solid oxide fuel cell (SOFC) sealing glass (YSO77), both in the as-cast and short-crystallized forms. MnO has been chosen as a "trapper" due to its potential ability to react with Cr, a major source for degradation of SOFCs. In the as-cast glass, both the glass transition and softening temperature decreased with increasing MnO. In the short-crystallized glasses, the softening temperature generally decreased with increasing MnO, and the presence of softening point suggested remaining glassy phases after short crystallization. The effect of MnO on the coefficient of thermal expansion (CTE) showed no distinct trend. Elastic properties of Young's and shear modulus decreased with increasing MnO for both the as-cast and short-crystallized glass.. The elastic properties in the crystallized glasses were generally higher than those of their as cast counterparts. The density of the glasses decreased with increasing MnO content, and was lower in the crystallized counterparts when compared to the as-cast glasses. The CTE seemed to fluctuate in both sets of glasses without and logical pattern. These fluctuations are still not understood and may require further investigation if the CTE of these glasses becomes a primary concern. But since MnO was chosen for its chemical interest and not its physical properties for this study these are not of general concern. The properties of the new glasses are adequate to be used in further SOFC applications, and the glass with 6 mol% MnO will be further investigated under actual conditions as a sealant.

Synthesis and Characterization of Nickel Oxide and Vanadium Oxide Nanostructures. *Amanda Tiano (University of Tennessee, Knoxville, TN); Stanislaus S. Wong (Brookhaven National Laboratory, Upton, NY).* In the field of nanotechnology, transition metals have attracted research interest because of their electronic and magnetic capabilities. Nickel(II) oxide (NiO), an antiferromagnetic material and p-type semiconductor, has been a large focus in research due to its diverse applications, such as catalysis, electrochromic devices, gas sensors, and magnetic films, making it an attractive material for the fabrication of nanodevices. Vanadium oxide (VO_2), like NiO, has also appealed to researchers because it undergoes a semiconductor-to-metal transition, known as a Mott transition, at 68°C. Below this temperature, VO_2 maintains monoclinic (M) phase and above converts to the metallic (R) phase. Since the Mott transition is also associated with reversible changes in optical, magnetic, and electrical properties as well as structure, VO_2 is also appealing for a variety of applications from energy storage to catalysis. NiO nanocrystals formed with and without the presence of a surfactant via thermal decomposition at 830°C in molten NaCl of two different precursors: nickel(III) oxide (Ni_2O_3) and nickel oxalate ($NiC_2O_4 \cdot 2H_2O$). These precursors formed quasi-circular particles measured as 283 ± 135 nm and 287 ± 100 nm in size, respectively. NiO nanosheets, 355 ± 166 nm in diameter and 44 ± 12 nm in thickness, were also formed by hydrothermal treatment of nickel acetate, $Ni(CH_3COO)_2 \cdot 4H_2O$, to generate nickel hydroxide with subsequent thermal decomposition at 400°C. Monoclinic VO_2 (B) nanorods, (width: 185± 37 nm, length: 1.4 ± 0.4 μm) were synthesized by hydrothermal treatment of V_2O_5. Subsequent thermal decomposition at 340°C in air produced V_2O_5 nanoparticles (107 ± 26 nm) while heating at 500°C in a flowing argon atmosphere produced larger monoclinic VO_2 (M) rod-like particles (width: 0.98 ± 0.38 μm, length: 6.8 ± 2.2 μm). The products were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and a magnetic property measurement system (MPMS). We have confirmed the presence of nickel oxide and vanadium oxides with XRD and determined size, shape and morphology with SEM. We have begun analysis with the MPMS but the current signal intensity associated with our samples has thus far been too weak for adequate interpretation. Through characterization, we can conclude the successful synthesis of nickel oxides and several forms vanadium oxide nanostructures by both molten salt and hydrothermal methods.

Semiconductor-Nanoparticle-(Poly)Ionic Liquid Composite. *Veronika Vajdova (University of Chicago, Chicago, IL); Millicent Firestone (Argonne National Laboratory, Argonne, IL).* The design, synthesis, and characterization of a hierarchically ordered composite whose structure and optical properties can be reversibly switched by adjustment of solvent conditions are described. Semiconductor cadmium sulfide (CdS)-nanoparticle containing ionic liquid-derived polymers were synthesized in a single step by UV irradiation of a Cd^{2+}-ion-precursor-doped, self-assembled ionic liquid gel, 1-decyl-3-vinylimidazolium chloride. Several approaches to preparing semiconductor ionic liquid derived polymer composites have been studied. In the first approach, a Cd^{2+} impregnated polymer is swollen in ethanol (EtOH), and then immersed in a sodium sulfide (Na_2S) solution to generate the encapsulated CdS nanoparticles. In the second approach, the Cd^{2+} impregnated polymer is directly swollen in a methanol-Na_2S solution. Both procedures yield transparent polymers with a light yellow to orange color. The yellow color, after photopolymerization of the binary mixtures with UV irradiation for two hours, indicates formation of CdS nanoparticles. The properties of the *in-situ* synthesized CdS nanoparticles were determined by UV-Vis and photoluminescence spectroscopy. Optical spectroscopy of the CdS-polymer composite shows a dramatic increase in the absorption at 450 nm. Moreover, this CdS impregnated polymer exhibits a fluorescence band located at 473 nm, whereas the control polymer showed no distinct bands. The polymerization described in this paper yields a material with improved mechanical properties, which can be both readily processed and applied in technologically relevant applications and environments.

Microbiologically Influenced Corrosion in Oil Pipelines: Causes and Effective Treatments. *Brenden Van Slyke* (*State University of New York at Stony Brook, Stony Brook, NY*); *Brenden Van Slyke* (*Brookhaven National Laboratory, Upton, NY*). Microbiologically Influenced Corrosion (MIC) is a serious problem in the oil industry. During oil extraction, especially secondary recovery, water may become mixed with the oil. This water can harbor microorganisms, that when not controlled can cause serious corrosion due to their metabolic processes. Two particularly damaging and widespread forms of bacteria involved in MIC are sulfate reducing bacteria (SRB) and acid producing bacteria (APB). The initial phase of this project was literature research into the causes and mechanisms for MIC. Then this information was applied to examining photographs and replicas of a pipeline suffering from possible MIC. Based on this examination, information on different treatment options were researched. The literature research aspects of the project were undertaken using electronic media such as the Internet and online journals, correspondence with industry officials and scientific researchers, and printed material. Literature research into the causes of MIC led to two distinct areas of interest. The first of these was the human factor, which dealt with poor maintenance and improper use of biocides allowing bacteria to proliferate in the system. On the bacteria side, the SRB and APB formed colonies with themselves and other non-corrosive bacteria. The bacteria can influence corrosion by the production of corrosive metabolites, hydrogen sulfide in the case of SRB and organic acids with APB present. SRB also can stimulate the development of iron sulfide deposits, which increase corrosion by enlarging the cathode. Furthermore, acids produced by APB can also complex with metal ions at the anode, removing them and accelerating the corrosion reaction. The replica casting were made from actual corroded oil pipe using Struers Repliset kits. These were then sputter coated using an Anatech Hummer VII to show more contrast. After examining the photographic evidence and replicas three types of corrosion were suspected. Two cases of MIC, one each by SRB and APB, were determined. One sample showed inorganic carbon dioxide corrosion and a fourth section exhibited an undetermined form of corrosion. The most important factor in limiting MIC is keeping oil pipelines clean and as water free as possible. Biocides containing glutaraldehyde as the active agent and quaternary ammonium compounds to break up biofilms are suggested. This project is part of a larger investigation into oil pipeline corrosion, which is ongoing.

Effect of Chemistry Variations on the Microstructure and Mechanical Properties of Creep Strength Enhanced Ferritic Steels. *Keely Wilson* (*Michigan Technological University, Houghton, MI*); *John Shingledecker* (*Oak Ridge National Laboratory, Oak Ridge, TN*). Grades 91, 92, and 122 steels (9–12% Chromium) are known as Creep Strength Enhanced Ferritic steels. These grades of steel are finding increased usage in the pressure retention components of advanced fossil energy systems (Ultrasupercritical Steam Boilers, Heat Recovery Steam Generators, etc.) because of their superior performance in high temperature, high stress environments. The chemical specifications for these grades are very broad, which may affect the mechanical properties and long-term performance of the alloy in service. Ideally in the normalized and tempered condition, Gr 91, 92, and 122, will have fully martensitic structures with no ferrite forming. In an earlier study, two compositions of each grade were produced by varying the amounts of austenite formers (C, Mn, Ni, N) and ferrite formers (Si, Cr, Mo, V, Nb) within the current specification range. These chemistry changes were guided by computational thermodynamics to alter the intercritical temperatures, the temperatures at which steel changes phase, and to cause the formation of ferrite under standard processing conditions. In this study, the mechanical properties of these samples were evaluated, and are compared to literature results for commercially produced material. High temperature (650°C) creep tests were run with loads ranging from 100 to 140 MPa for times exceeding 1,000 hours. Tensile tests were run at both high temperature (650°C) and room temperature (25°C). Digital Imaging software was used to analyze the steel microstructures to determine the amount of martensite and ferrite present in each alloy. It was found that both the tensile strength and the creep strength of the alloys decreased substantially with the presence of ferrite in the material. This critical finding clearly shows that the specification range for these alloys is too broad which may result in commercially produced materials with properties far from expectations. A limited evaluation of thermodynamic predictions and microstructural findings was also conducted. The data collected in this study, combined with data from other tests, such as long term creep tests, thermo-mechanical simulation, and thermodynamic modeling will later be used to create more specific standards for Gr 91, 92 and 122 alloys.

Synthesis of Calix[4]cyclohexanol to Serve as an Electron Trap. *Benjamin Zalisko* (*Elmhurst College, Elmhurst, IL*); *John A. Schlueter* (*Argonne National Laboratory, Argonne, IL*). Electrons are known to be trapped between positive charges of liquid water and alcohol molecules without being incorporated into the molecules' electron shells. Seeking to trap an electron within a single molecule, calix[2]cyclohexanol should be synthesized by hydrogenation of calix[4]arene at 200 psi and 90°C, resulting in calix[2]cyclohexanone. This should be followed by reduction with $NaBH_4$ to produce calix[2]cyclohexanol. Using an electron donor (Na metal or electrolysis), an electron should become trapped between the partially positive, tetrahedral hydrogen atoms of the internal hydroxyl groups of calix[2]cyclohexanol. This trapped electron's presence will be confirmed by its ability to retain magnetic spin with electron spin resonance spectroscopy. Acting as a capacitor, this electron trap will retain electron spin efficiently and could lead to further studies of conduction, electron spintronics, and even new media for micro processing.

Cadmium-doped SiO_2 Nanoparticle Label for the Electrochemical Immunodetection of Protein IgG. *Xiaohai Zhang* (*University of Washington, Seattle, WA*); *Jun Wang* (*Pacific Northwest National Laboratory, Richland, WA*). Semiconducting nanoparticles have garnered enormous attention in the field of biosensor development within the past decade. Due to recent advancements in the control of their growth size and shape, nanoparticles such as silica MCM-41, which have unique structural functionalities, are now an applicable material in creating novel methods of bio-detection. In this report, an amperometric biosensor using biochemically modified silica nanoparticles was developed for the detection of protein IgG via sandwich immunoassay. This novel electrochemical immunosensor is based on the encapsulation of Cadmium ions within mesoporous aluminosilicate nanoparticles (SiNPs) MCM-41. The synthesized silica NPs — antibody conjugates were then characterized with electrochemical detection as well as UV spectrum analysis. Preliminary tests have confirmed that Cadmium ions were immobilized inside the mesoporous shell of the silica NPs; they've also shown that the protein antibodies were successfully conjugated. In these experiments, the performance of the electrochemical immunosensor was evaluated via carbon Screen Printed Electrodes (SPE), and a detection sensitivity of 10 ppm was achieved. This report has shown that SiNPs has great potential for future research and development of electrochemical bio-sensors; however, optimizations in its synthesis method are still required to increase the detection limit. Cadmium labeled silica NPs offers a viable approach for the rapid, simple, and cost-effective analysis of biological samples.

Medical and Health Sciences

Determining the Optical Properties of Biological Tissue Samples Using an Integrating Sphere Method. *Marcus Allegood* (*North Georgia College and State University, Dahlonega, GA*); *Justin S. Baba* (*Oak Ridge National Laboratory, Oak Ridge, TN*). Wavelength dependent light interaction with biological tissue can be described using three parameters: the scattering and absorption coefficients and the cosine of the average scattering angle (g). To accurately determine these optical properties for different types of tissue at specific wavelengths would be beneficial for a variety of different biomedical applications. The goal of this project was to take a user defined g-value and determine the remaining two parameters for a specified range of wavelengths. In order to collect the needed data for all the wavelengths in a timely and accurate manner, a fully automated computer program and process was developed. Using a single integrating sphere method, scattered light intensity inside the sphere was recorded via a spectrometer as either transmitted or reflected light from the tissue sample. LabVIEW was used to write programs to collect raw intensity